4. Digestions

4.1 Microwave Digestions

4.1.4 Nitric acid and hydrogen peroxide (30 %): peat, bone char, plant material (hardly digestible)

Dana Zimmer, Rhena Schumann

Suitability

This method of digestion can be preferably used for peat, bone char and plant material that is difficult to digest, such as wood, roots and potato tubers. If bone char and similar material is digested, the microwave program with longer digestion time should be selected (see below). All important elements (e.g. nutrient) are generally measurable e. g. Fe, Mn, Al, Na, Ca, K, Mg and P. For simultaneous trace element analyses, e. g. Cd, Cu, Pb, Zn, all vessels have to be cleaned by acid.

If individual bone char particles (< 0.03 g) are digested and measurement of, for example Fe and Zn, is planned besides P, the sample should be digested by only 1.7 ml HNO_3 and 1 ml H_2O_2. After digestion the extract should be filled to only 20 or 50 ml to exceed the quantification limit of trace elements for determination at ICP-OES. It could be possible that in this case, the extract has to be diluted for P-determination.

Photometric detection of P is unusual. Due to nitric acid, the detection with molybdenum blue is not possible (Hansen & Koroleff 1999, chapters 4.1.2 and 5.2.3).

Concentration range

The measurement range and limit of detection for P strongly depend on selection of the detection method. Generally, the ICP-OES is the method with the highest (that means worst) limit of detection and quantification (chapter 5.1). However, it is possible to increase material weigh-in and the amount of extraction agent to adjust the concentration in the measurement solution to achieve the measurement range of the instrument.

Generally, the ICP-OES can detect all P compounds in the measurement solution and not only "free" phosphate. However, this might not cause any differences because strong digestion conditions converted almost all P compounds to phosphate. An advantage of the ICP-OES is the wider measurement range than those of photometrical methods (chapter 9), which can decrease potential errors by dilution.

Mainly essential nutrients (e.g. Fe, Mn, Al, Na, Ca, K, Mg and P) are determined in extracts of plant material, peat and bone char.

Protocol

Day 1: Preparation

- Put on your protective clothing (gloves, coat, glasses).
- Weigh-in ca. 0.1 to 0.5 g fine milled material into Teflon vessels of microwave (note precise mass).
- Place standards (chapter 6.6) and 2 blanks (5 ml HNO_3 and 3 ml 30 % H_2O_2) per run in the microwave.
- Add 5 ml conc. HNO_3 under the fume hood (clean inner vessel wall from sample material) and 3 ml 30 % H_2O_2.
- Set vessels with soil samples with acids open under the running fume hood overnight.

Day 2: Digestion

- Close vessels, mark blanks and dissolved standards as "empty place" (if possible, in the microwave), operate microwave according to the instructions (see below), cool down for around 1 h.
- Transfer extraction solution via (plastic) funnel in 50 or 100 ml (plastic) volumetric flask (Fig. 4.1.2-3). The solution has to be clear but may be green or yellow coloured.
- Rinse microwave vessel and funnel with ultra-pure water into the volumetric flask and fill flask with ultra-pure water to 50 or 100 ml. This volume has to be exact because from this volume the element concentration is calculated.
- Filter (e.g. Macherey-Nagel™ folded filter papers MN 612 retention 5-8 µm or phosphorus-poor MN 616 G retention 4-12 µm) the extraction solution into (acid-rinsed) polyethylene bottles (reference sample).
- Fill around 20 ml of solution into "ICP-vessels".

Table 4.1.4-1 Digestion program for microwave MarsXpress for plant dry matter and peat

Level	Max. Power (W)	Power (%)	Ramp (min)	Temperature (°C)	Holding (min)
1	1200	100*	15:00	200	5:00
2	1200	100	1:00	210	5:00
3	1200	100	1:00	220	5:00

Table 4.1.4-2 Extended Digestion program for bone char and similar hardly digestible material

Level	Max. Power (W)	Power (%)	Ramp (min)	Temperature (°C)	Holding (min)
1	1200	100*	15:00	200	5:00
2	1200	100	1:00	210	5:00
3	1200	100	1:00	220	**45:00**

* Settings for "Power" depend on numbers of sample-filled vessels: 8-12 vessels (50 %), 13-20 vessels (75 %) and > 20 vessels (100 %).

Day 3: Measurement

- Determination of P at ICP-OES (wavelengths for P 214,914 or 213,617 nm, chapter 5.1)

Reference

Hansen H P, Koroleff F (1999) Determination of nutrients. In: Grasshoff K, Kremling K, Ehrhardt M (Eds.) Methods of seawater analysis. Wiley-VCH, 159-251, DOI: 10.1002/9783527613984.ch10

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